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**Long life durability of electrodes of electrical resistance change
method for damage monitoring of CFRP composite structures**

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14. ABSTRACT

Investigated the applicability of the electrical resistance change method through experiments of CFRP laminates [16-18]. Instead of using two or four electrodes, this research adopted multiple electrodes mounted on the surface of the target structure to identify delamination location and dimension. Orthotropic electric conductivity was also measured experimentally for three types of fiber volume fractions, and the paper revealed that electric conductivity in thickness direction of CFRP was approximately one thousandth of conductivity of fiber direction. The large difference brings difficulty of identifications of delamination location from the measured electrical resistance changes. For these studies, a response surface method was adopted as a tool to solve inverse problems delamination location and dimension were obtained from measured electric resistance changes of multiple segments between electrodes. The previous study [20] used FEM analyses of various delamination lengths and locations of beam-type specimens to investigate the reason of the large estimation errors of delamination locations; furthermore an improvement to obtain higher estimation performance was proposed, which used the data normalization method before making response surfaces. The normalized method provides highly precise estimations of location and size: 7mm error in size and 10mm error in location experimentally for CFRP laminates. Other researchers attached electrodes on the edges of the specimens [1-15]. The method does not cause damages of the electrical contact at the electrodes during loading. The method, however, does not monitor the location of the damages instead. The important point is to mount multiple electrodes on the surface of the target CFRP structure in our method although mounting electrodes on the surface may cause degradation of electrical contact during loading. In the present study, long life durability of the electrodes made on the surface of CFRP is focused on. Several types of making electrical contact are attempted here; silver paste: electrical conductive adhesive and copper plating. Surface treatment of copper plating after polishing specimen surface is investigated in detail experimentally. A static tensile test is performed to measure electrical resistance change during the tensile test. A strain gage is attached on the surface to obtain the relationship between the stiffness change and the electrical resistance change. After the static tensile test, cyclic loading test is performed to investigate long life durability of the electrodes. Four-probe method is adopted in the present study to measure electrical resistance changes.

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1. Introduction

The vertical tail wing of Airbus A300 of the flight AA587, which crashed in November 12th in 2001, is made from CFRP (Carbon Fiber Reinforced Plastics) composites, and the delamination was one of the causes found from the fragments of the tail wing. The delamination of CFRP composite structures is very difficult to inspect visually, and damage is difficult to detect even for ultrasonic inspection due to the high damping and complicated microstructure of the composites. This motivates the development of a new monitoring system of delaminations for CFRP composites.

Recently several researchers have adopted an electric resistance change method to detect internal damages such as fiber breakages, delaminations and matrix cracks in CFRP laminates [1-15]. They adopted two or four-electrode method to measure the electric resistance of entire specimens, and they found existence of damage from the change of the measured electric resistance. In contrast to fiber-optic sensors, the electric resistance change method does not require expensive instruments for measurements. Since the method adopts reinforcement carbon fiber itself as sensors for damage identifications, this method does not cause reduction of strength; it is applicable to existing structures merely by means of attaching multiple electrodes on the target CFRP structures.

Our research group has investigated applicability of the electrical resistance change method through experiments of CFRP laminates [16-18]. Instead of using two or four electrodes, we adopted multiple electrodes mounted on the surface of the target structure to identify delamination location and dimension. Orthotropic electric conductivity was also measured experimentally for three types of fiber volume fractions [19], and the paper revealed that electric conductivity in thickness direction of CFRP was approximately one thousandth of conductivity of fiber direction. The large difference brings difficulty of identifications of delamination location from the measured electrical resistance changes.

For these studies, a response surface method was adopted as a tool to solve inverse problems: delamination location and dimension were obtained from measured electric resistance changes of multiple segments between electrodes. The previous study [20] used FEM analyses of various delamination lengths and locations of beam-type specimens to investigate the reason of the large estimation errors of delamination locations; furthermore an improvement to obtain higher estimation performance was proposed, which used the data normalization method before making response surfaces. The normalized method provides highly precise estimations of location and size: 7mm error in size and 10mm error in location experimentally for CFRP laminates. Other

researchers attached electrodes on the edges of the specimens [1-15]. The method does not cause damages of the electrical contact at the electrodes during loading. The method, however, does not monitor the location of the damages instead. The important point is to mount multiple electrodes on the surface of the target CFRP structure in our method although mounting electrodes on the surface may cause degradation of electrical contact during loading.

In the present study, long life durability of the electrodes made on the surface of CFRP is focused on. Several types of making electrical contact are attempted here; silver paste: electrical conductive adhesive and copper plating. Surface treatment of copper plating after polishing specimen surface is investigated in detail experimentally. A static tensile test is performed to measure electrical resistance change during the tensile test. A strain gage is attached on the surface to obtain the relationship between the stiffness change and the electrical resistance change. After the static tensile test, cyclic loading test is performed to investigate long life durability of the electrodes. Four-probe method is adopted in the present study to measure electrical resistance changes.

2. Materials and experimental methods

2.1 Specimens

Material used in the present paper is unidirectional CFRP prepreg. The type of the unidirectional prepreg sheet is P3060-15 (T300/3601) produced by Toray Co. Ltd.. Using the prepreg, unidirectional $[0_4]_T$, cross-ply laminates of $[0_2/90_2]_S$ and quasi-isotropic laminates of $[0/90/\pm 45]_S$ were fabricated. The fiber volume fraction is approximately 0.5. Thickness of the laminates is approximately $t=0.5\text{mm}$. Cure condition is $120^\circ\text{C} \times 1.1\text{MPa} \times 20\text{min} + 155^\circ\text{C} \times 1.1\text{MPa} \times 120\text{min} + 180^\circ\text{C} \times 1.1\text{MPa} \times 120\text{min}$ produced using an electrical furnace. From the rectangular plate, tensile specimens as shown in Fig.1 were made.

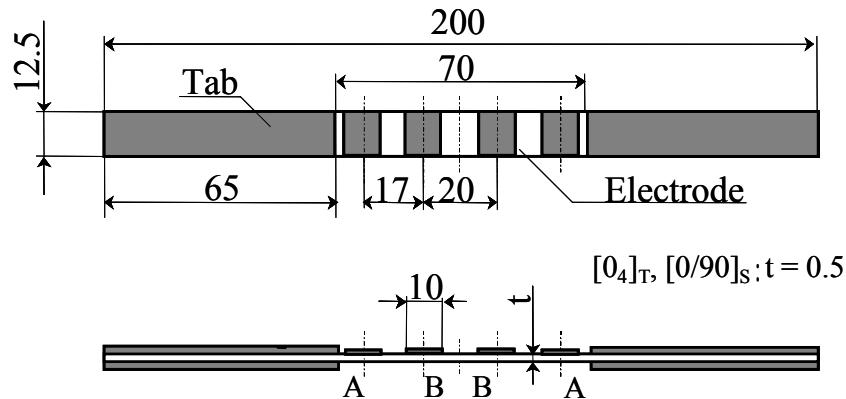


Figure 1 Specimen configuration

2.2 How to make electrodes

The process to make an electrode is as follows.

(1) Do polishing with sand paper. Use rough sand paper (JIS #240)

Use masking to prevent from polishing other area as shown in Fig.2.

Polish until the black powder of crashed carbon fibers is recognized in the entire area.



Fig.2 Polishing

(2) Clean up the polished surface with acetone

(Do not touch the surface with naked fingers)

(3) Remove the epoxy resin with concentrated sulfuric acid and hydrogen peroxide. The concentrated sulfuric acid and hydrogen peroxide make persulfuric acid. The persulfuric acid does not give damage to the carbon fibers but may give significant damage to epoxy resin. Figure 3 shows the several drops of hydrogen peroxide. Wait 30 seconds or more to remove the sizing around carbon fibers. The waiting time depends on the acid resistance of the resin and cure condition. This must be decided by trial and error.



Fig.3 several drops are enough

(4) Wash the surface with enough water

(5) Clean up the polished surface with acetone

(6) The processes from (3) to (5) are cycled several times if the removal of sizing around the fibers is not enough.

(7) Mask the unpolished area

(8) Attach the direct current supplier to the specimen (CFRP is used as a minus electrode and a copper plate is used as a plus electrode)

(9) Dip the both electrodes into the solution of copper sulfate

(10) Direct current is applied (see Fig. 4)

Condition:

Copper sulfate: 240g/(1000cc)

97% concentrated sulfuric acid: 49g/(1000cc)

Direct current: (First step) $2\text{A}/\text{dm}^2$ ($\text{dm}=10\text{cm}$) $\times 10\text{min}$
 (Second step) $6\text{A}/\text{dm}^2 \times 10\text{min}$

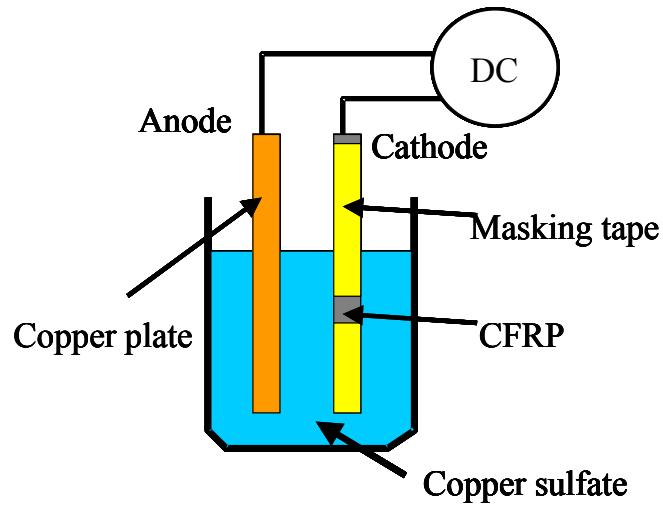


Fig. 4 Copper plating method

(11) Lead wires of enameled wire of diameter of 0.1mm are soldered to the electrodes as shown in Fig. 5.



Fig. 5 Electrode and lead wire

2.3 Test methods

Electrical resistance of a specimen is measured with a four-probe method: the outer couple of probes are used to apply electric current and the inner couple of electrodes are used to measure electrical voltage. To measure the electrical resistance, a commercially available LCR meter produced by Hioki Co. Ltd. is used here.

Single cycle tests and cyclic tests are performed. For the tests, a MTS hydrostatic test machine was used. Short-term cyclic tests were performed at 15 Hz at the condition that the maximum load of the cyclic loading is approximately from 60 to 95 % of the fracture load. Long-term tests were performed at 25 Hz at the condition that the maximum load of the cyclic loading is approximately 20% of the fracture load.

Since the initial electrical resistance is unstable, the electrical resistance at the 1000 cycles was

decided as initial resistance in the present study.

3. Results and discussion

3.1 Single cycle test

Figure 6 shows the results of the static tensile test of unidirectional specimen in the elastic deformation region. The abscissa is the applied strain and the ordinate is the applied stress and the measured electrical resistance change. As shown in this figure, the electrical resistance is completely linear relationship in the elastic deformation region.

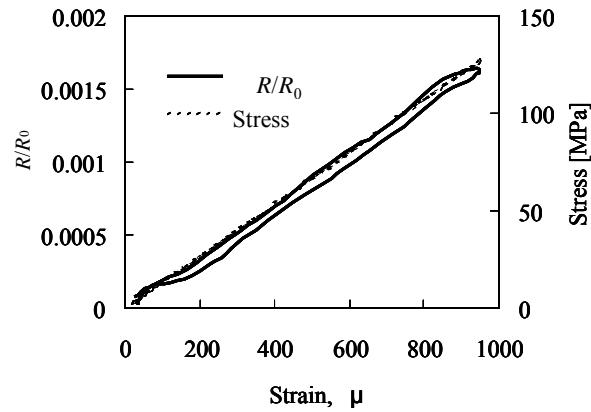


Fig. 6 Single cycle test of unidirectional specimen

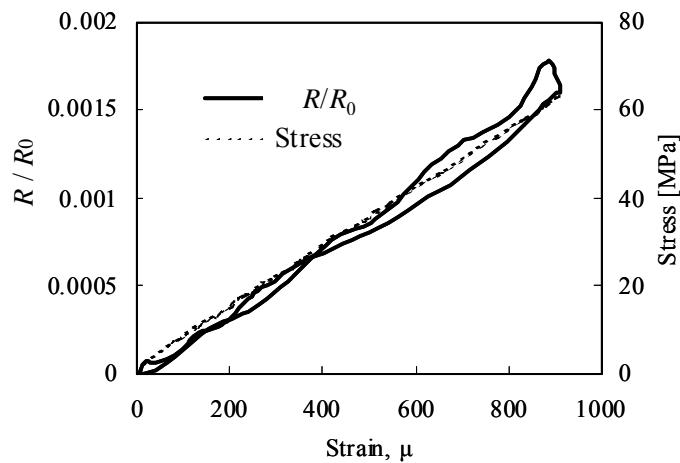


Fig. 7 Single cycle test of cross-ply specimen

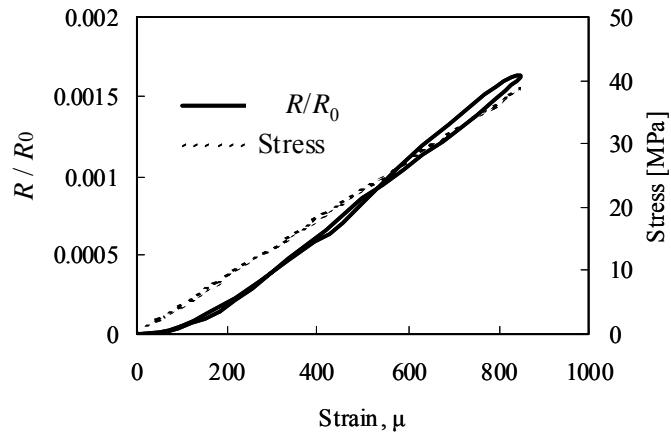


Fig. 8 Single cycle test of quasi-isotropic specimen

Figure 7 and 8 are the single cycle tests of cross-ply laminate and quasi-isotropic laminate test specimens in the completely elastic deformation region. These figures show that the electrical resistance change increases with the increase of applied tensile strain using the four-probe method regardless of the stacking sequences.

3.2 Tensile fracture tests

Figure 9, 10, 11 are the tensile fracture test results. The abscissa is the applied strain and the ordinates are the electrical resistance changes and the applied mean stress. Up to the tensile strength, the electrical resistance can be measured without debonding of the electrodes. As shown in these figures, electrical resistance increase just before fracture can be measured for the quasi-isotropic laminate. These figures show the copper plating electrodes are reliable method.

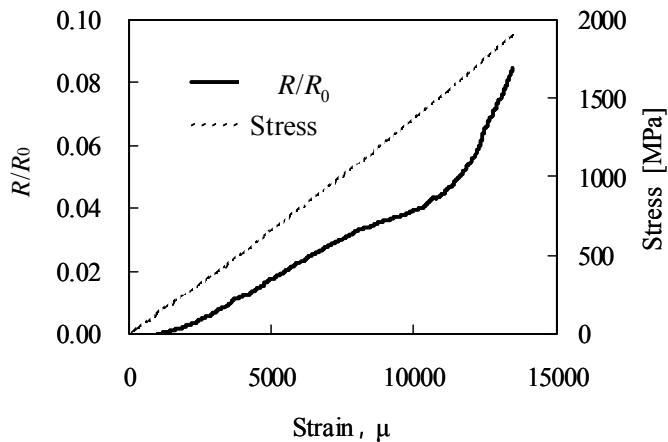


Fig. 9 Electrical resistance increase of tensile fracture test of unidirectional laminate

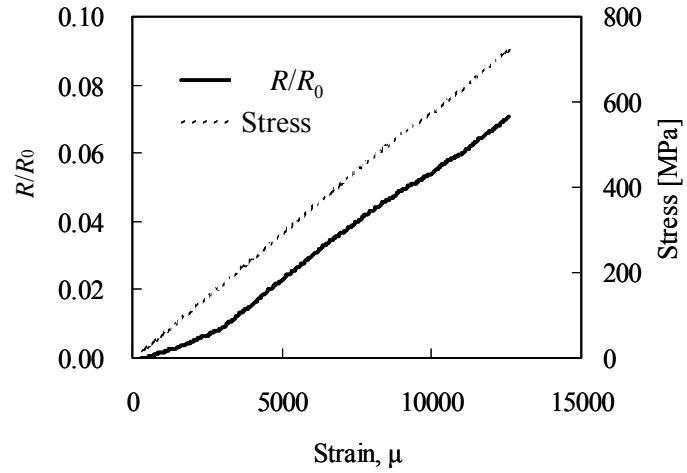


Fig. 10 Electrical resistance increase of tensile fracture test of cross-ply laminate

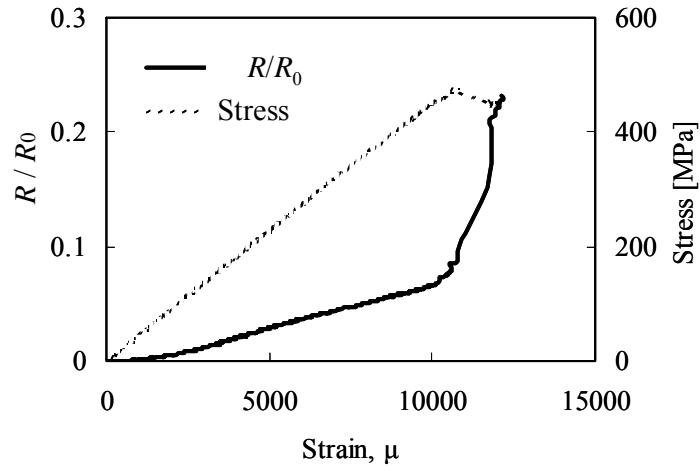


Fig. 11 Electrical resistance increase of tensile fracture test of quasi-isotropic laminate

3.3 Fatigue loading tests

Fatigue tests were performed using unidirectional laminates and cross-ply laminates to measure fatigue limit of the CFRP laminates. Figure 12 shows the results of the fatigue tests. The abscissa is the number of cycles to failure and the ordinate is the applied stress normalized by average tensile strength. The solid circle symbols show the results of the unidirectional laminates and the open square symbols show the results of cross-ply laminates. For quasi-isotropic laminates, only a long term fatigue test performed to save experimental time.

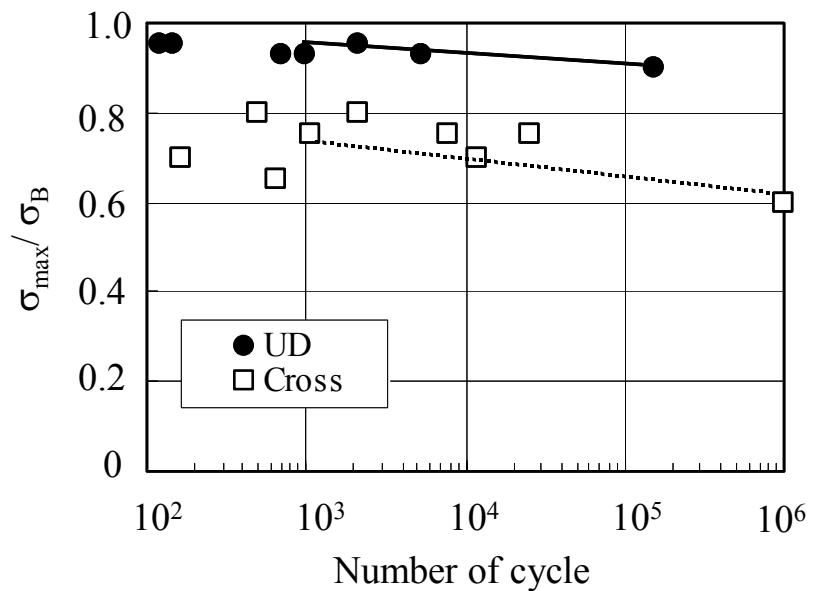


Fig. 12 Fatigue cycle test results.

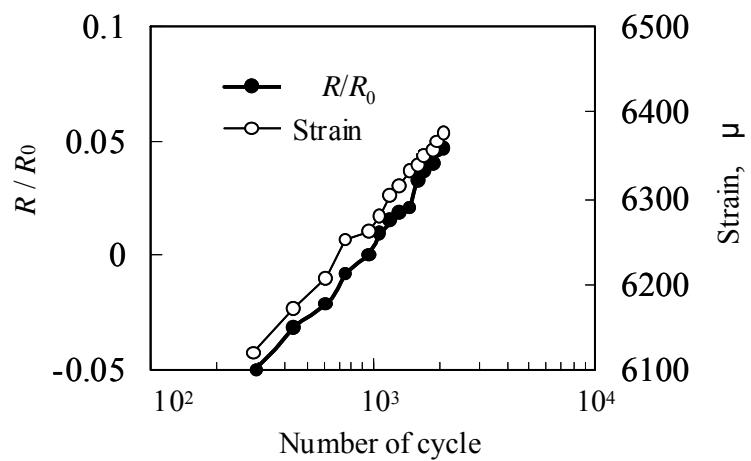


Fig. 13 Short term fatigue test result of unidirectional laminate

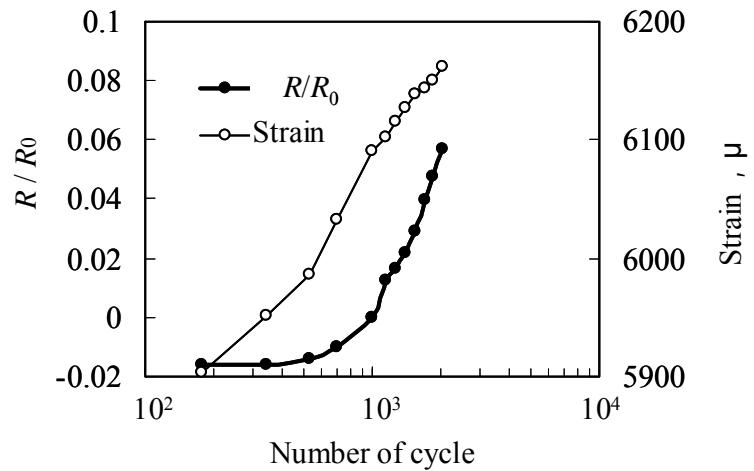


Fig. 14 Short term fatigue test result of cross-ply laminate

Figures 13 and 14 show the results of short term fatigue tests (fatigue life is short). The abscissa is the number of cycles and the ordinates are the electrical resistance change and the applied strain. As shown in these figures, electrical resistances can be measured up to the failure of the specimens even for the cyclic loading test. These figures show that the electrodes are reliable even for the high stress short term cyclic condition.

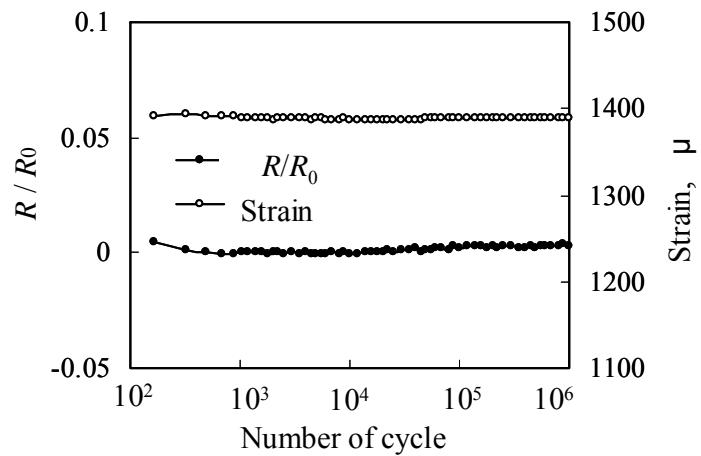


Fig. 15 Long term fatigue test result of unidirectional laminate

The maximum applied load is set to 20 % of the static tensile strength and long term cyclic tests (small cyclic load test) were performed for the three types of the specimens. Figure 15 shows the results of the unidirectional specimen. The abscissa is the number of cycles and the ordinate is the electrical resistance and applied strain measured at the average load of the cyclic stress. The solid circles show the electrical resistance changes and the open circles are the applied strain. As shown in this figure, there is almost no change in the electric resistance up to 10^6 cycles.

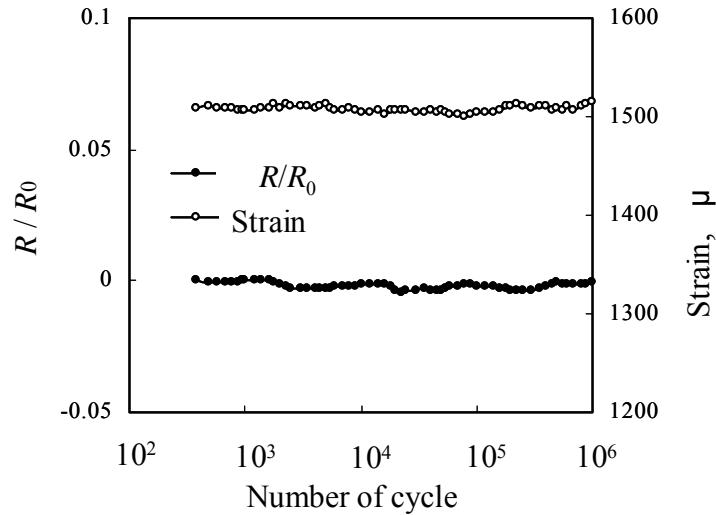


Fig. 16 Long term fatigue test result of cross-ply laminate

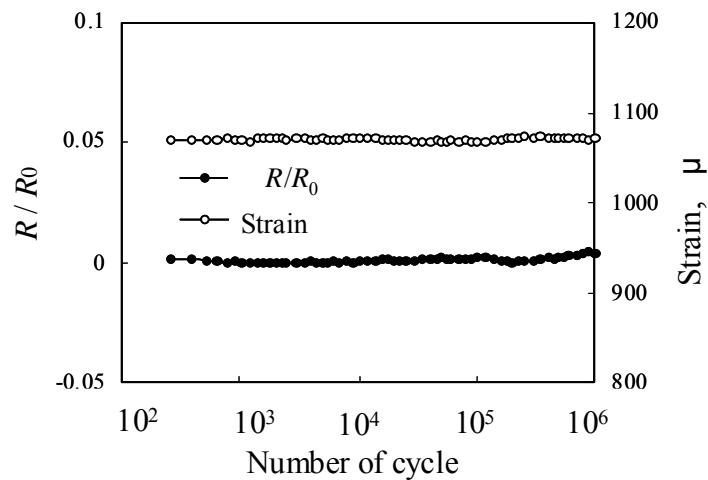


Fig. 17 Long term fatigue test result of quasi-isotropic laminate

Figure 16 and 17 show the results of the cross-ply laminate and quasi-isotropic laminate. These figure also show that the there is almost no change in the electrical resistance even for the long term cyclic tests. The copper plating method is useful for any types of stacking sequences.

Cross sectional observations were performed at the electrodes to observe the damage of electrodes. Figure 18 shows the typical cross sectional view of the electrode. For every types of the specimen before cyclic tests (no previous load is applied), debonding of the copper electrode is observed just under the soldering position. This seems that the soldering heat causes the debonding between the copper and the CFRP. After cyclic tests, significant change was not observed. This debonding may not affect the electrical resistance change but the debonding of copper electrode should be avoided for practical applications with temperature changes.

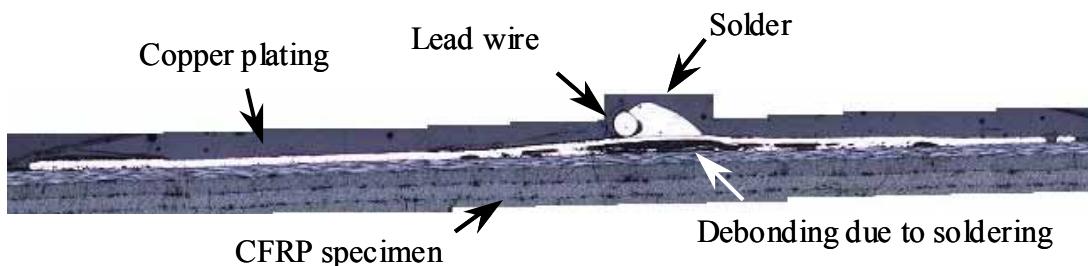


Fig. 18 Cross sectional view at the electrode

To prevent the debonding caused by the soldering, integrated lead wire copper plating method is tried here. Figure 19 shows the results of the integrated lead wire. The lead wire is attached to the copper plated area and copper plating is performed again with the copper lead wire. The lead wire is connected by using the copper plating without using soldering. Using the new method, longer cyclic loading test will be performed in our future work.

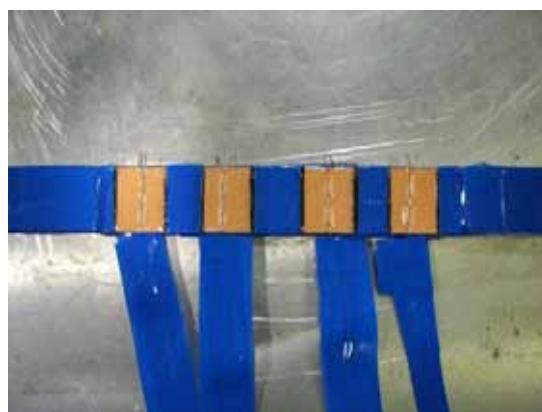


Fig. 19 Copper plating method to attach lead wire

4. Conclusions

The report explains how to make reliable electrodes and the results of long-term cyclic tests. The results obtained are as follows.

- (1) Copper plating method to make electrode is very reliable for short term and long term cyclic loading.
- (2) Copper plating method is applicable for any types of stacking sequences.
- (3) To prevent the debonding caused by soldering, integrated copper plating with lead wire is recommended.

7. Future works

- (1) Longer cyclic test must be performed with the new integrated lead wire.
- (2) Compression tests should be performed to investigate the compression fracture.

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